14p.

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September 21, 1973

(NASA-CR-120265) REPORT ON SKYLAB -M552 SAMPLES (Battelle Columbus Labs., Ohio.) ~~20~~ 19 p HC $4.00 CSCL 11F

N74-28988

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REPORT ON SKY LAB-M552 SAMPLES
CONTRACT NO. NAS-8-28725

This report summarizes the work conducted on M552 samples at Battelle's Columbus Laboratories since July 18.

MATERIAL TRANSFER

The review meeting held at MSFC was attended on July 18. Specimens SLS-1 and SLS-3 were hand carried to Battelle following that meeting. Specimens SLN-2 and SLN-4 (after sectioning) were picked up by Mr. Pattee at ORNL on August 20. SpecimenSLN-4 was delivered to the University of Wisconsin on August 21. Specimen SLS-3, after sectioning at Battelle, was given to the University of Wisconsin on August 2.

SPECIMEN PROCESSING

Processing of the specimens at Battelle has followed the procedures previously developed and demonstrated in processing ground characterization samples.

Specimens SLS-1 and SLS-3 were attached to a helium mass spectrometer at one end of the tube. A rubber stopper was inserted in the opposite end of the tube. Tests

were then run for leakage through the annular braze joint. No leak was detected in SLS-1. Leakage occurred in SLS-3 as expected. The mass spectrometer used had previously been calibrated to 1.08×10^{-11} atm sec.

Both specimens were then centered in a lathe for marking of sectioning planes. Centering was with a tapered rod on the ID of the tube. SLS-3 was marked routinely. However, complete circumferential scribe marks could not be made on SLS-1. A few measurements were then made to determine the extent of offset or out of roundness. The tube showed 0.003-in. TIR at the igniter end and 0.005-in. TIR at the opposite end. TIR on the sleeve ranged from 0.010 to 0.013 in. An additional longitudinal scribe line was added to the sleeve to assist in subsequent orientation. This line is at an angle to the original groove, being closest at the igniter end.

Radial cuts were then made to section the specimens into thin washer like pieces. The locations of these cuts is tabulated in Table 1. Also indicated are longitudinal cuts made to expose ring groove areas.

Cut sections of Specimens SLS-1 and SLS-2 were mounted and polished for detailed examination.

VISUAL EXAMINATION

All sections were examined at low and high power magnification to determine (1) visual appearance of the braze, (2) confirm radiographic findings, and (3) select sections for metallographic examination. Results are discussed below.

TABLE 1. LOCATION OF SECTIONING PLANES - SKYLAB SPECIMENS-M552

Specimen SLS-1[a]		Specimen SLN-2[b]		Specimen SLS-3[a]		Specimen SLN-4[b][c]	
No.	MM.	No.	MM.	No.	MM.	No.	MM.
	-4		-8		0		
1.1	0	2.1	-4	3.1	3		
1.2	3	2.2	0	3.2	10		
1.3	10	2.3	2	3.3	24		
1.4	14	2.4	5	3.4	32		
1.5	19	2.5	10	3.5	35		
1.6	23	2.6	15				
1.7	31	2.7	20				
1.8	35	2.8	25				
1.9	39	2.9	31				
1.10	43	2.10	35				
		2.11	38				
		2.12	42				
1.3.1	3.5/10	2.5.1	10.5/15				
0 to 180 degrees		90 to 270 degrees					
1.7.1	23.5/31	2.9.1	31.5/35				
30 to 210 degrees		90 to 270 degrees					

(a) Sectioned at Battelle
(b) " at ORNL
(c) Data available from ORNL or University of Wisconsin

Specimen SLS-1

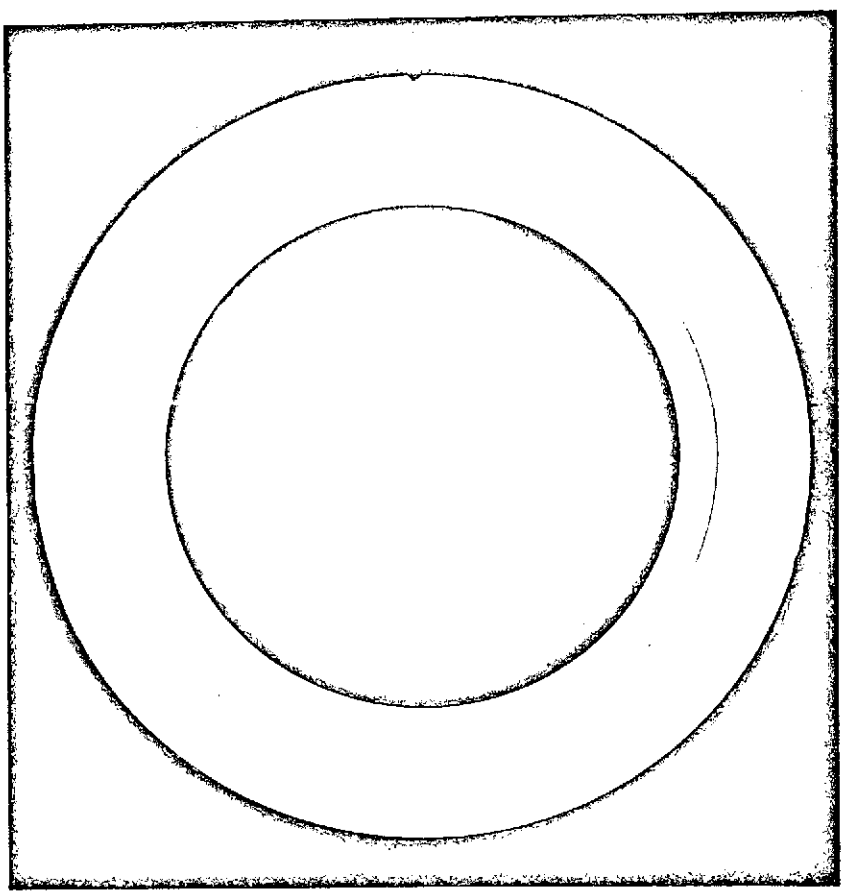
The tube-sleeve assembly was almost a perfectly void free braze from end to end. Braze filler metal was evident around the entire joint circumference in each section. The only void area apparent in the radiograph (just below the top ring groove at Position F) was exposed in Section 1.7.1. Excess filler metal flowed through the center slits and collected inside the tube between about 80 degrees and 235 degrees, and 270 degrees and 10 degrees. The ring grooves were generally empty of filler metal except for a small bridge in the igniter end groove at Position B.

The most notable observation was wide variations in the braze gap of all sections between the ring grooves. This is discussed in more detail later.

Figure III-1[(a)] shows the appearance at 3 mm (1.2). This section is between the igniter end of the sleeve and the adjacent ring groove. Sections at 0, 31, and 35 mm were similar in appearance. Some idea of the gap variations is evident: compare the thickness at 45 degrees and 235 degrees. A very small void is present at 235 degrees.

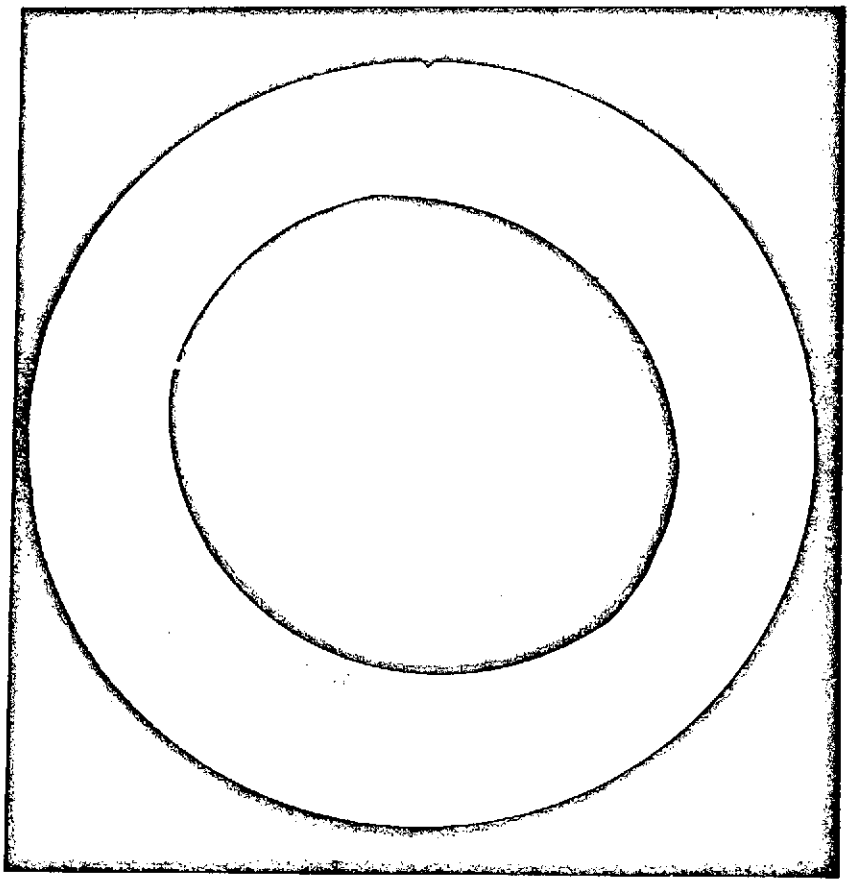
Figure III-2 shows the section at 14 mm and was generally typical of other sections between the ring grooves at 19 and 23 mm. Both the variation in gap width and braze alloy inside the tube are seen readily.

(a) Figure numbering is intended to fit into final report.



4X As Polished 7G867

FIGURE III-1. SECTION SLS-1.2(3mm)



4X As Polished 7G868

FIGURE III-2. SECTION SLS-1.4(14mm)

The longitudinal sectionsof the igniter end ring groove are shown in Figure III-3. Excellent fill of the gap ırea has occurred. Also, fillets have been retained both at the ends of the gap and in the remote corners of the ring groove. Portions of the excess filler metal inside the tube are also seen. Some narrowing of the joint gap at the 10-mm plane is apparent.

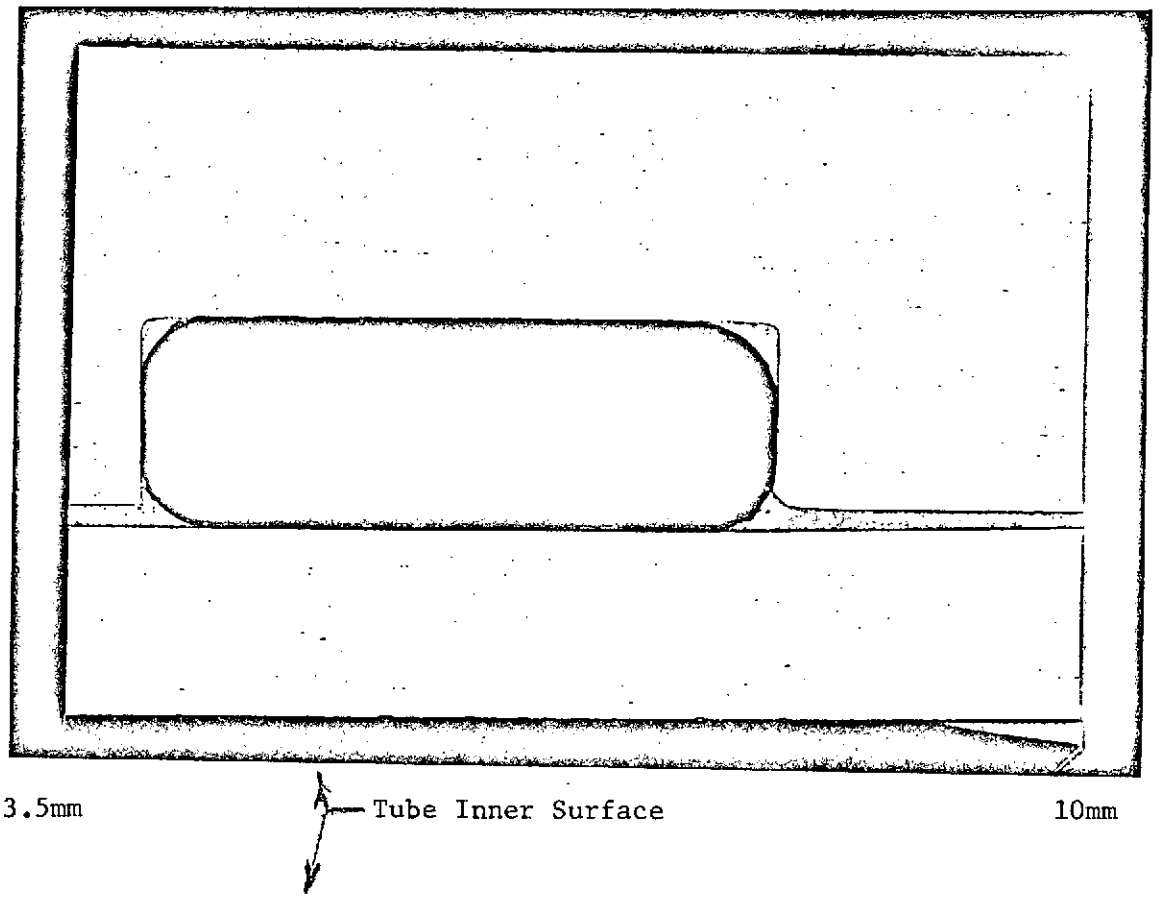
The longitudinal section at 30 degrees between23.5and 31 mm is shown in Figure III-4. This section was taken through the only area containing an apparent void on the radiograph. The void area is confirmed. Again, good fillets were retained in the ring groove except where insufficient alloy was available next to the void. Higher magnification views of the void region show the typical appearance of a shrinkage void caused by draining of some of the final liquid to other regions during final solidification.

Microstructures

The braze region microstructures as shown in Figure III-5 are 250 and 500X. The phases present are essentially identical to those observed in the ground characterization specimens.

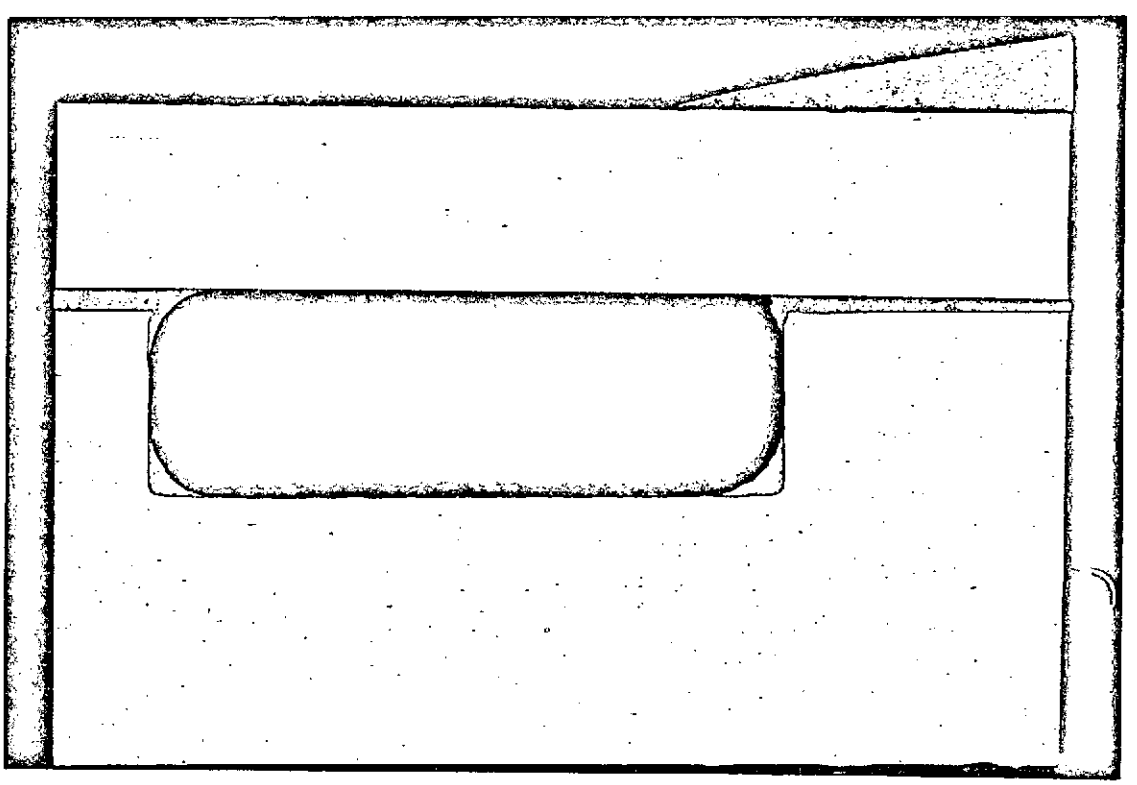
Microstructure of the bulk braze alloy that solidified inside the tube is also similar to comparable regions in ground characterization specimens as shown in Figure III-6.

Some regions of the sleeve-tube interface where the gap was very tight are shown in Figure III-7. These regions were examined in some detail to determine if a solid-state weld existed. Figure III-7b shows the same region after etching and discloses evidence of grain growth across the interface needed to substantiate that solid-state welding (diffusion bonding) did indeed occur.



0°

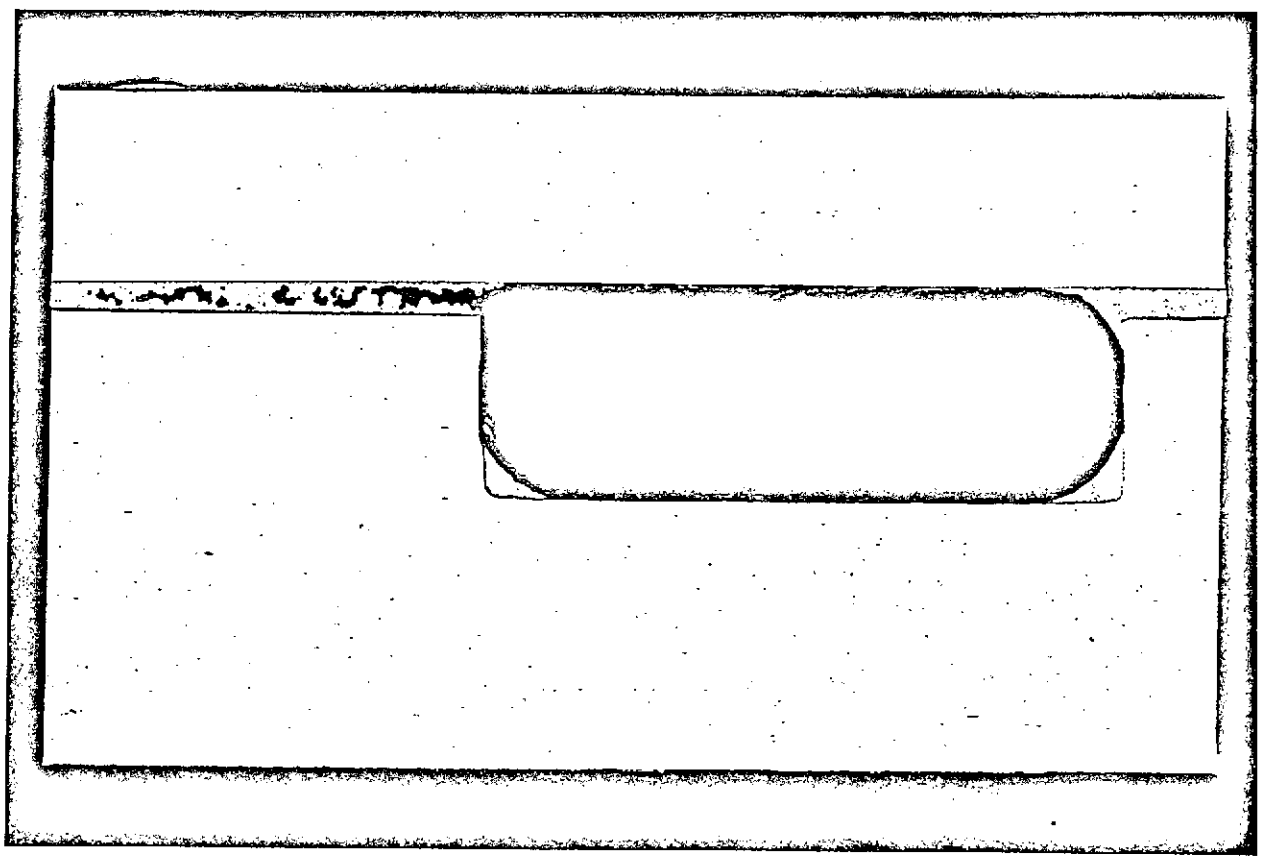
3.5mm — Tube Inner Surface — 10mm



180°

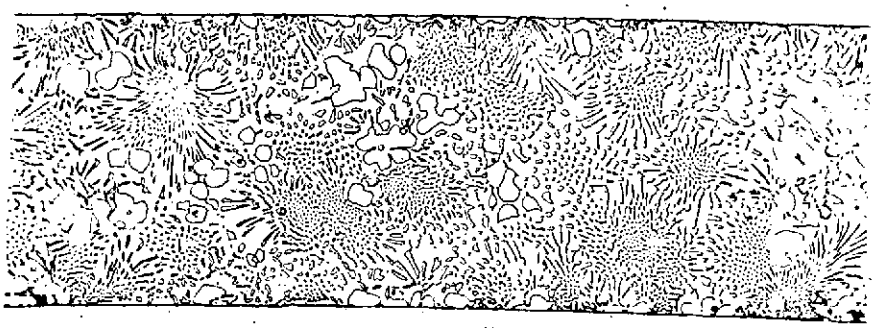
20X As Polished 7G869 7G870

FIGURE III-3. SECTION SLS-1.3(3.5 to 10mm)

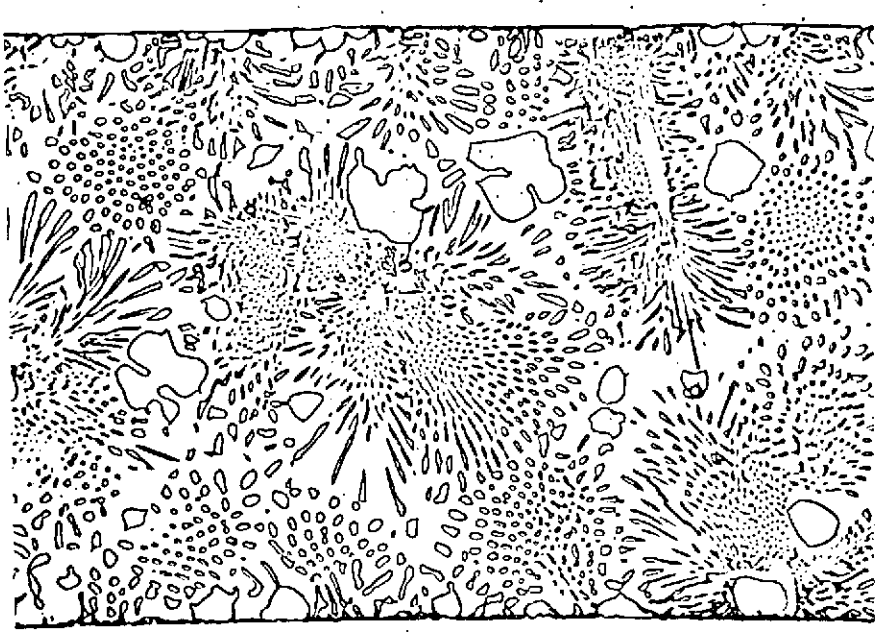


20X As Polished 7G871

FIGURE III-4. SECTION SLS-1.7.1(23.5 to 31mm & 210°)

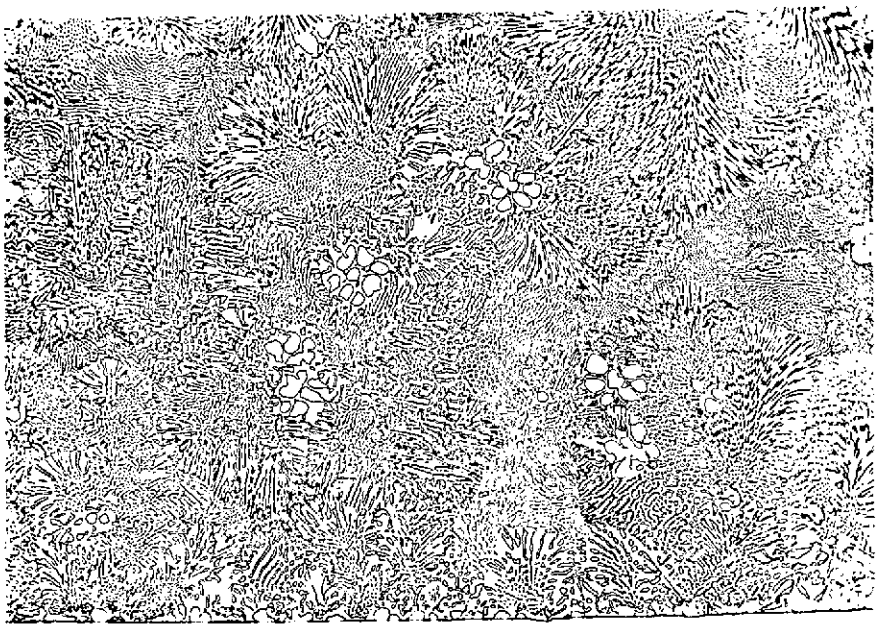


250X As Polished 7G853

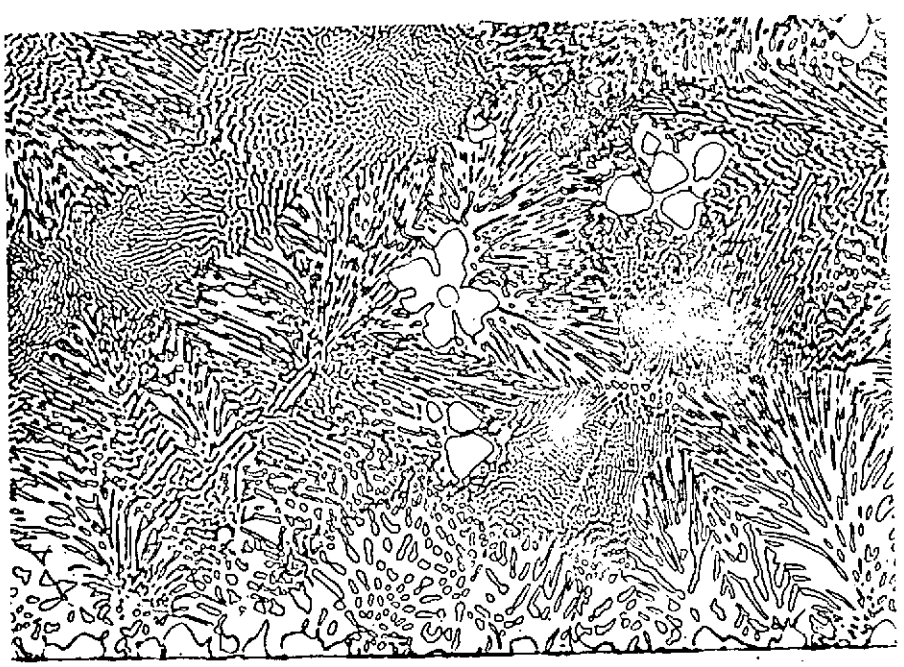


500X As Polished 7G856

FIGURE III-5. SECTION SLS-1.4(14mm)(300°)



250X As Polished 7G860



500X As Polished &G862

FIGURE III-6. SECTION SLS-1.4(14mm-300°)

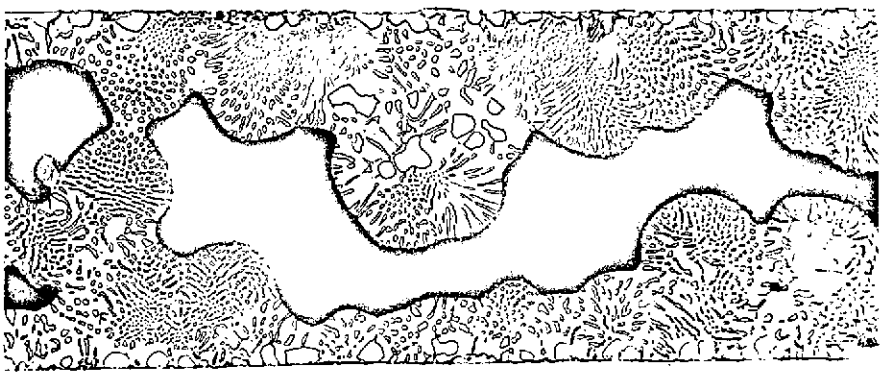
a.

500X As Polished 7G851

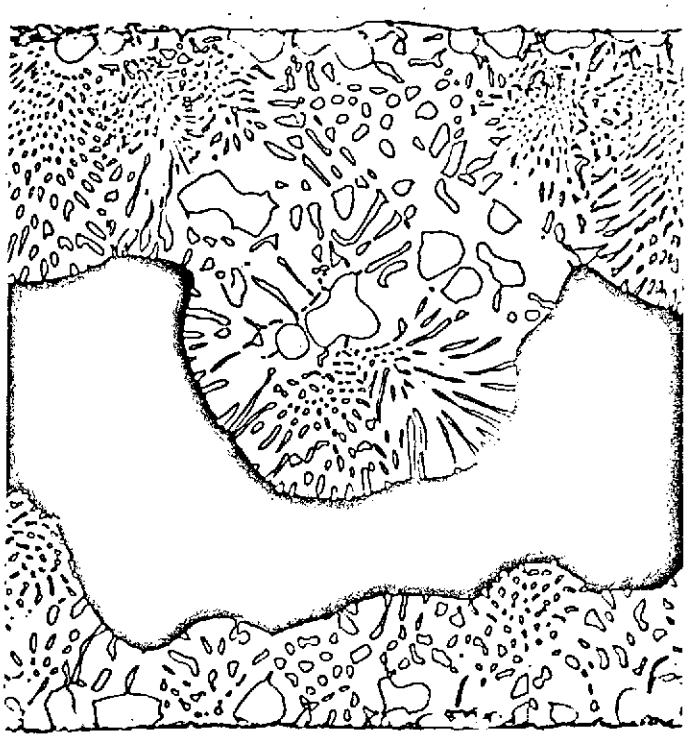
b.

500X 97HCL,3HNO_3,0.5g$CuCl_2$ 7G852

FIGURE III-7.. SECTION SLS-1.4(14mm-0°)



250X As Polished 7G864



500X As Polished 7G866

FIGURE III-8 SECTION SLS-1.7.1(24mm & 210

Microstructures in the area contain a void is shown in Figure III-8. The void shape is similar to that of comparable regions exained in ground characterization specimens. The voids are a result of draining of the area during the last stages of solidification.

Gap Variation Studies

Measurements of both the joint gap and diameters at various points were made in attempts to resolve the gap variations observed. The joint gap measurements are shown in Table III-2. The measured gap ranged from 0 to a maximum of 0.007 in., with most measurements being less than the intended 0.005-in. gap. Average gaps at each sectioning plane are plotted in Figure III-9, to better illustrate this variation. Diameter measurements of individual sections have indicated some minor out of round conditions, but have not been helpful in establishing the basic cause of the gap variations.

Specimen SLN-2

The tube-sleeve assembly contains a moderate amount of void area as reflected in the radiograph. Examinations are still in progress but the following significant finds have been made to date:

(1) This specimen also contains joint gap variations that are to some degree similar to those found in SLS-1. Joint gap measurements made to date are tabulated below.

Section	MM.	Gap thickness, inch, at position shown in degrees			
		0	90	180	270
2.3	2	.008	.006	.010	.010
2.6	15	.003	.003	.004	.008
2.7	20	.001	.002	.004	.006
2.8	25	.003	.003	.006	.008
2.10*	35	.004	.005	.004	.005

* Gap is quite uniform since this section contains a portion of the wedge.

The joint gap appears to be almost as expected at either end of the sleeve. The small gaps between the ring grooves appear to be related to tube-sleeve offset.

TABLE III-2. MEASUREMENTS OF JOINT GAP-SPECIMEN SLS-1

Sample No.	MM.[a]	Gap Thickness, Inch X 10^{-4}, at Position shown in degrees												
		0	30	60	90	120	150	180	210	240	270	300	330	Average
1.1 inner	0	33	6	5	8	3	3	4	2	3	4	4	9	5
outer	0		12	15	14	32	27	23	27	23	11	21	4	19
1.2[c]	3	42	34	30	36	48	50	52	57	58	55	55	50	47
1.3[c]	3.5	49						48						49
	10	38						28						33
1.4	14	0	6	8	0	0	11	25	18	9	50	54	19	17
1.5	19	1	0	2	0	1	9	12	9	19	47	38	11	12
1.6	23	25	8	4	23	29	11	9	36	64	51	31	25	26
1.7[c]	23.5		9						68					38
	31		33						70					52
1.8	35	38	39	42	47	53	45	47	54	57	54	48	44	47
1.9	39	0	0	4	5	5	4	5	3	6	11	9	13	5

(a) Location along length.

(b) This contains the wedge except at 0°, hence there are two gaps.

(c) Longitudinal section.

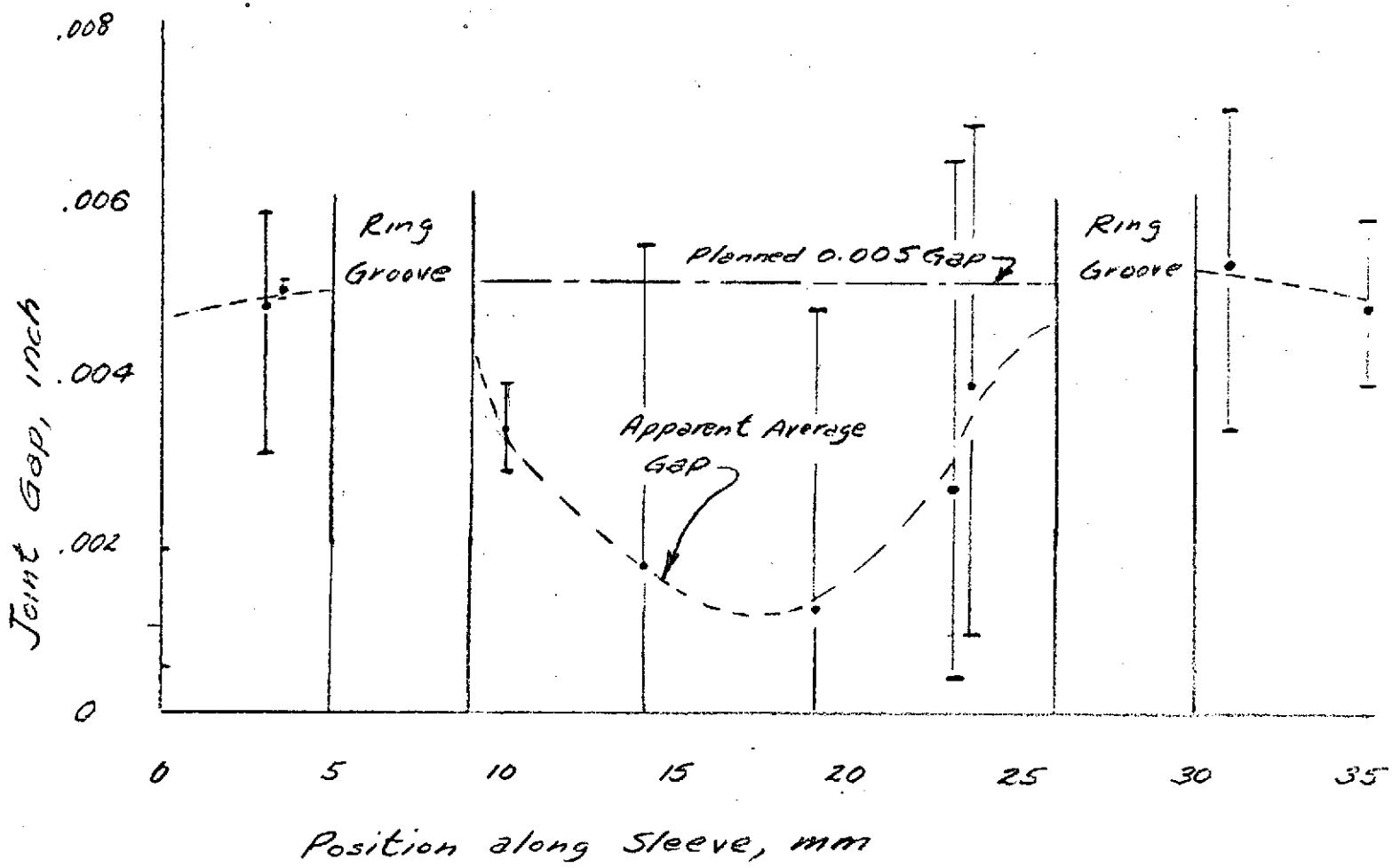


FIGURE III-9 PLOT OF JOINT GAP MEASUREMENTS

(2) Significant variations in the extent of reaction between the base metal and braze alloy appear at different locations. The amount of reaction phase adjacent to the base metal is generally greater where an appreciable joint gap exists.

(3) The braze alloy microstructure also varies appreciably, both within this specimens and in comparison to the microstructure observed in ground characterization samples. Three distinct microstructures have been noted:

(a) A structure essentially identical to the microstructure found typically in all ground characterization samples.

(b) A structure in which only a globular appearing second phase, believed to be copper-nickel, is in a matrix phase, believed to be silver. The amount of globular phase present is estimated at between 30 and 90 volume percent.

(c) A structuee in which a needle like second phase, apparently high in copper, is present in a matrix phase similar in appearance to the matrix described in (b) above. The needles are typically oriented perpendicular to the joint interface.

Although there appears to be a relation of these structures to joint thickness, this requires further study. Sections 2.3, 2.6, 2.7, and 2.8 all exhibit the three structures at various points around the circumference. Also, notable,

is the abruptness with which the microstructure changes from one type to another. Section 2.10 which is a very uniform gap entirely around at the end of the sleeve is entirely structure type a. Section 2.4, which is through the igniter ring groove, and is consequently a thick, more massive region is entirely structure Type b.

(4) A meniscus is evident in Section 2.5 (longitudinal through the ingiter ring groove). Significantly, this meniscus has an uneven surface similar to the surface of the shrinkage void regions. Comparison of this surface, with that shown in Adams report of July 16 (Figure 15) suggests that the absence of gravity has affected the meniscus contour.

CONCLUSIONS

Detailed examinations of specimen SLS-1 and SLN-2 have disclosed a number of differences from similarly processed ground characterization samples. These examinations are continuing and the results are being analyzed. Analysis at this point does not permit defining any specific effects to the zero gravity processing, althgouth it is likely that this may result from further study.